

Characterization of a Temperature Regulated Electronic Nicotine Delivery System for Potential use as a Reference Device

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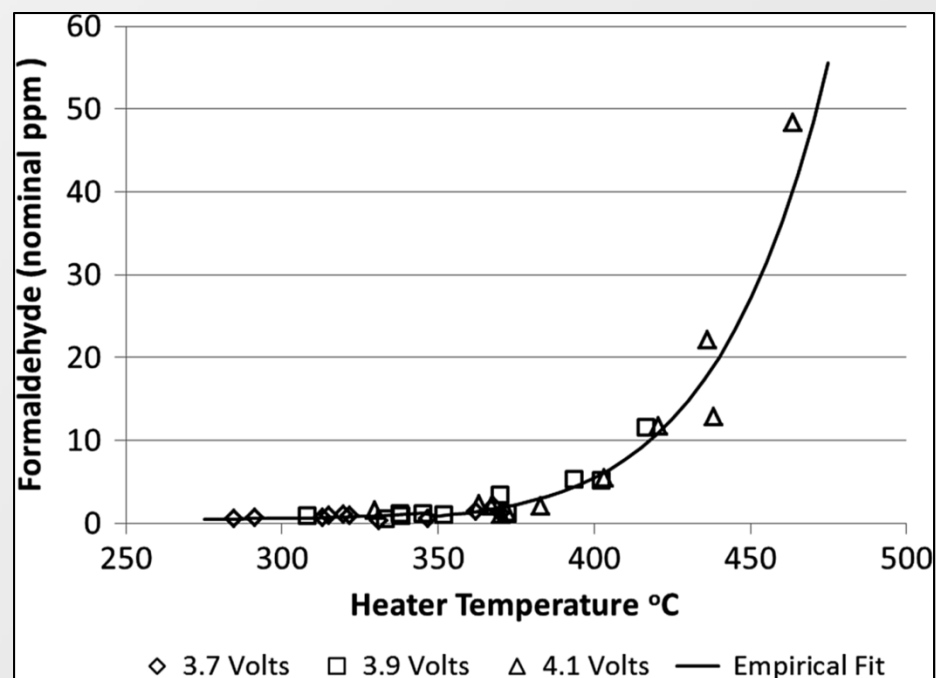
Why do we need a reference device?

- Method Development & Validation
 - Reference device
- Collaborative Study samples
 - r & R values
- Process monitor control
 - Routine sample analysis

Aldehyde Temperature Dependence

Upon heating of the e-liquid inside of the device tank, propylene glycol and glycerin are transformed into formaldehyde, acetaldehyde, and acrolein

The heat of the device coil exhibits an exponential relationship to the formation of the aldehydes, leading to extremely high productions of aldehydes at high temperatures



Data from: Flora et al. 2017

Evidence of Coil Overheating



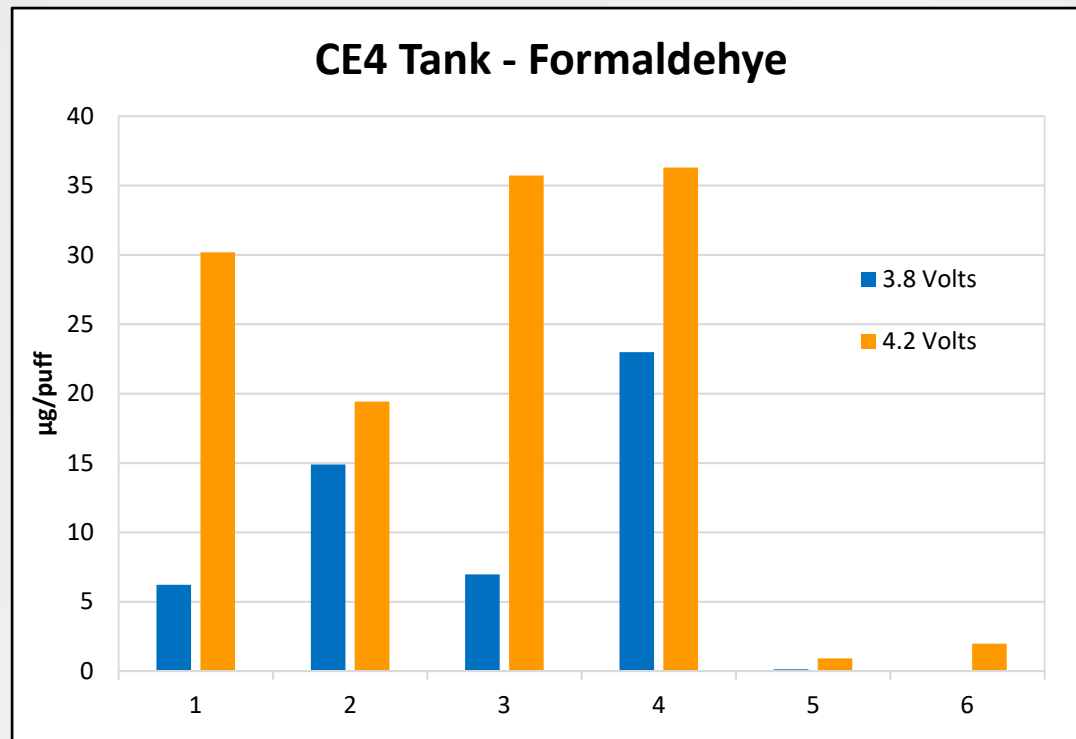
Unused coil



Used coil

Evidence of charring on wick
(example from CE4 tank)

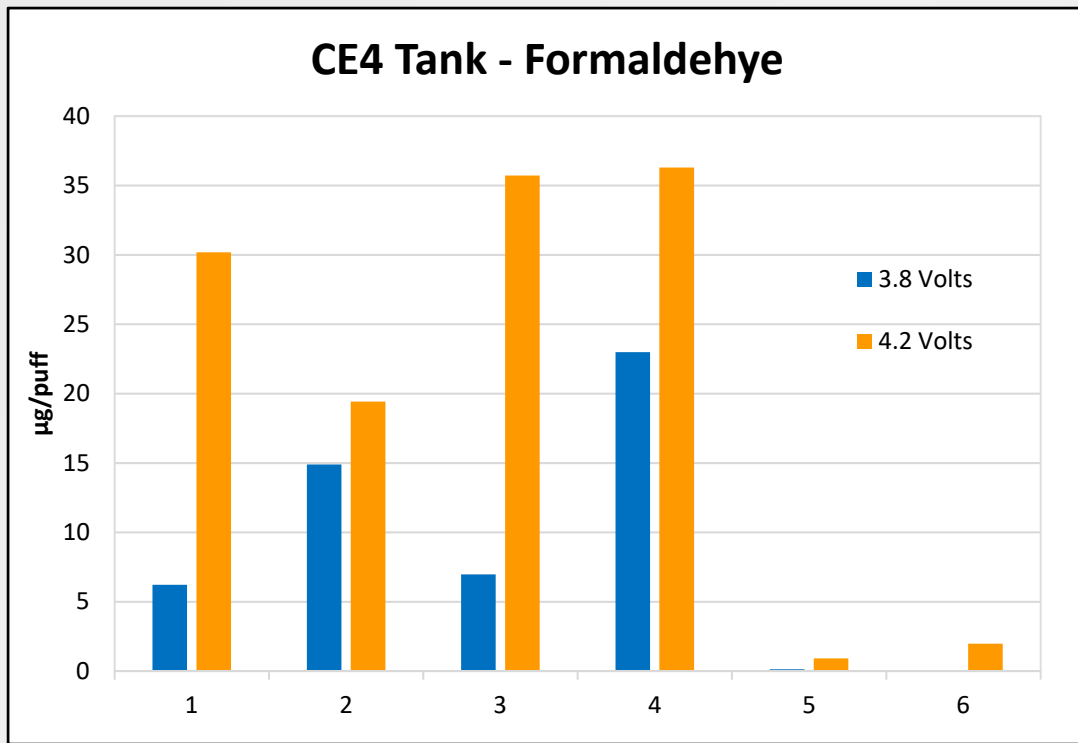
Variability



Six CE4 tank devices that do not have the ability to regulate coil temperature were tested

Two different battery voltages were tested, with the same number of puffs were taken from each puff block (25 puffs)

Variability



CE4	Formaldehyde µg/puff	Formaldehyde µg/puff
Rep	3.8 Volts	4.2 Volts
1	6.21663	30.1791
2	14.88963	19.4187
3	6.97368	35.721
4	22.98786	36.29871
5	0.1323	0.91434
6	0.07644	1.97274
AVG	8.55	20.75
SD	8.94	16.14
% RSD	104.56	77.78

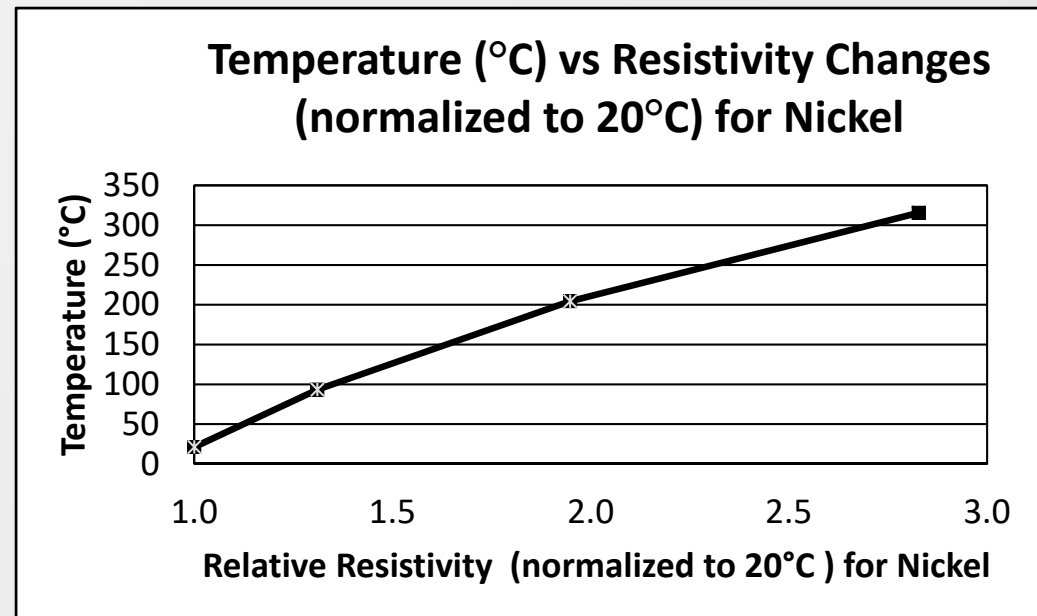
I. G., et al. (2016). "Effect of variable power levels on the yield of total aerosol mass and formation of aldehydes in e-cigarette aerosols." *Regulatory Toxicology and Pharmacology* 75: 58-65.

Temperature Regulated (TR) Devices

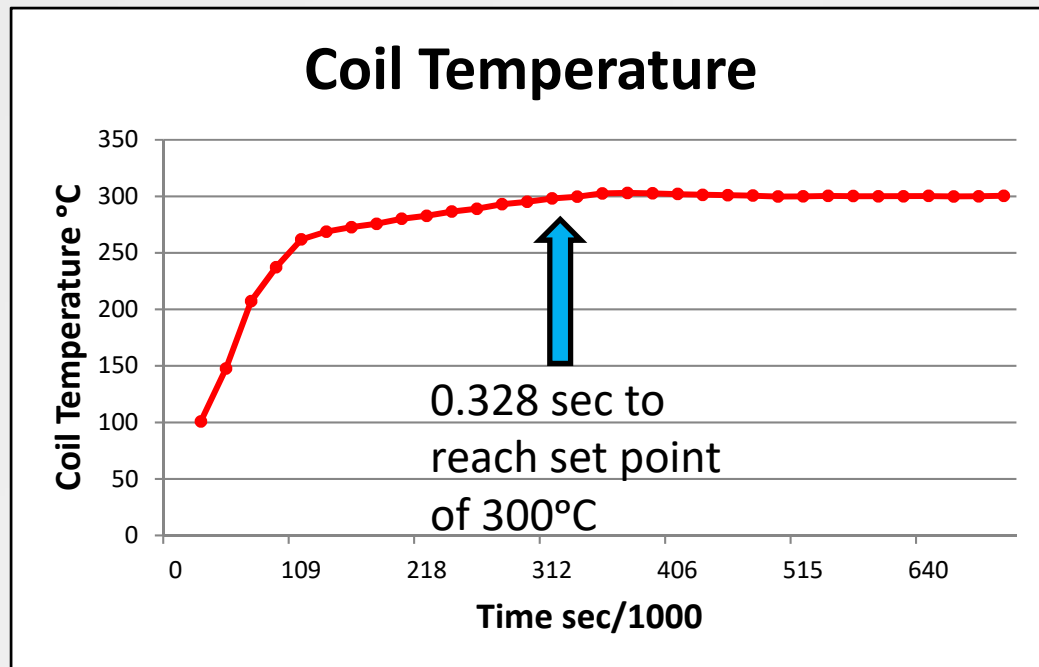
- A possible solution to device variability
 - Potential elimination of aldehyde overproduction
 - Occurs when either too much power or not enough liquid is supplied to the atomizer
 - The possible repeatability and reproducibility of devices generating Aldehydes
 - Native aldehyde production from an E-liquid base consisting of Propylene Glycol, Glycerin, and Nicotine

Temperature Regulated Devices

The relationship of the relative resistivity of the coil material to the temperature of the coil allows for accurate monitoring and control of the vaping temperature



Temperature Profile (Pre-Heat off)



Coil temperature over time of the activation of a temperature regulated device containing E-liquid

Temperature rises rapidly and directly to the set temperature (300 C) and maintains itself at this temperature over the full activation time of the device

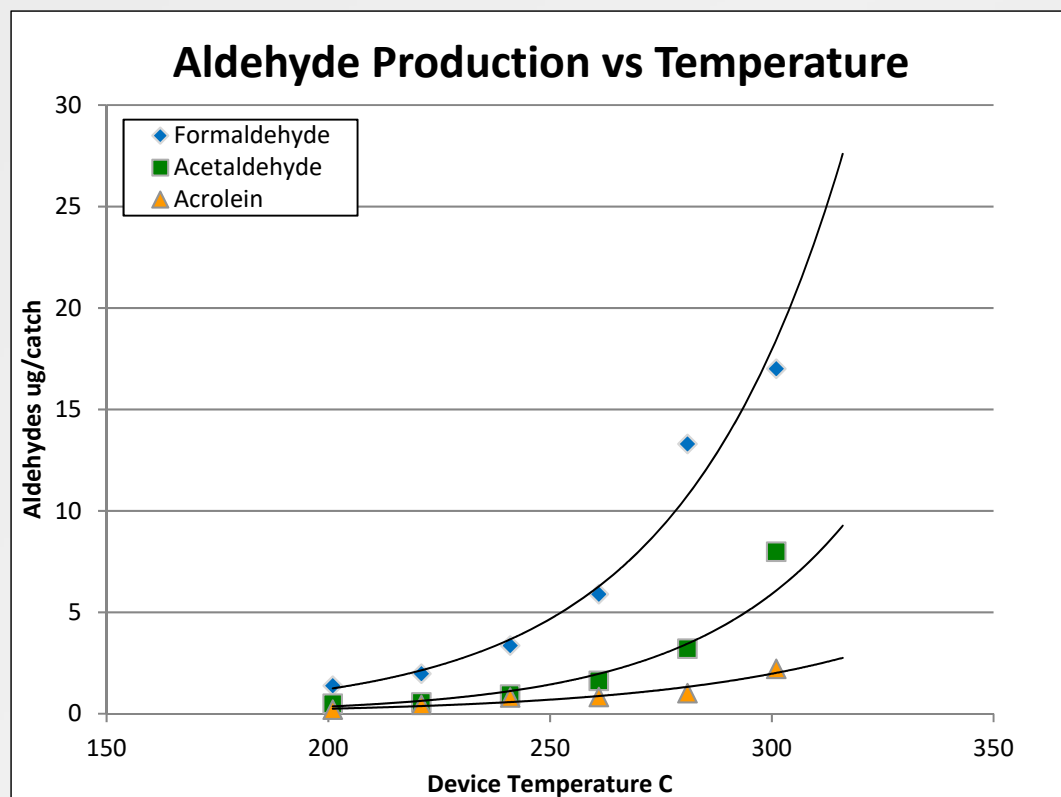
Device Characterization

- How much E-liquid does the device consume?
- What concentration of aldehydes is produced per gram of e-liquid consumed?
- Does the device produce the same amount of Aldehydes each time?

Analytical Methodology

- Aerosol was collected using an automatic “button pusher” using a 55mL constant flow puff over 4 seconds every 30 seconds
- E-liq: 50:50 PG/VG with 2% nicotine was used with all samples
- Samples were collected using new coils for each device, with the device pre-heat function off
- Aerosol samples were analyzed for aldehydes (formaldehyde, acetaldehyde, and acrolein)

Yield Profiles vs Temperature



Yield profile of a singular device was generated to determine optimal conditions for aldehyde production

Data was collected from 200-300 C

The device showed an exponential relationship of aldehyde production with increasing temperature

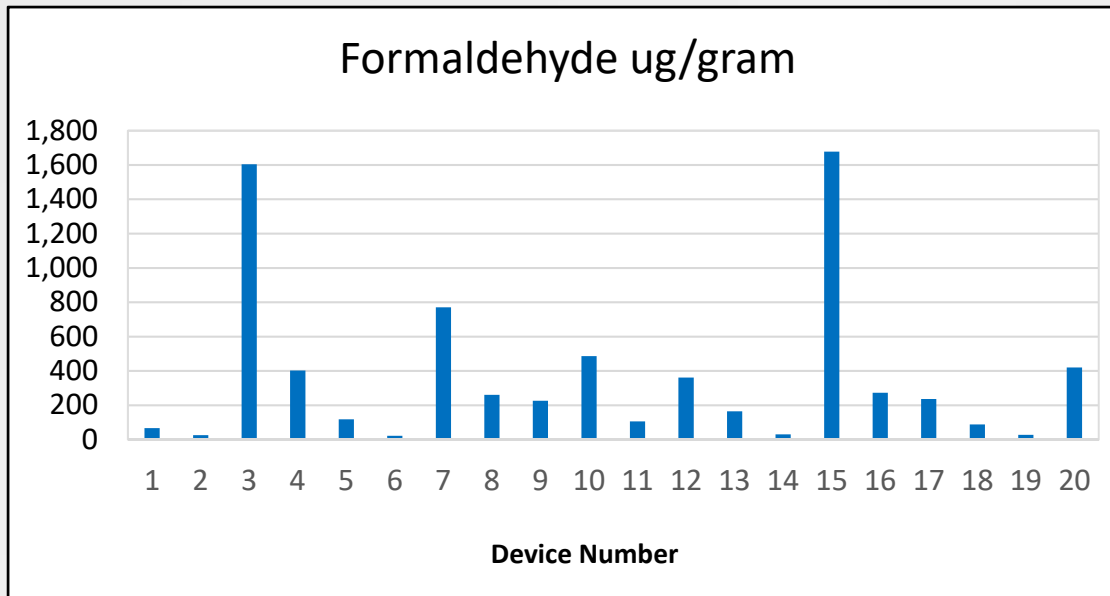
Temperature Determination

	Temperature °C	Formaldehyde µg/sample	Acetaldehyde µg/sample	Acrolein µg/sample
Band of relatively low-level aldehyde production	201	1.38	0.49	0.20
	221	1.97	0.56	0.44
	241	3.36	0.94	0.80
	261	5.83	1.62	0.82
Band of analytically relevant aldehyde production	281	13.3	3.20	1.01
	301	17.0	7.98	2.21

Experimental Design

- Native delivery at higher temperature
 - Device repeatability
 - Device reproducibility
- Fortified E-liquid delivery at a lower temperature
 - E-liquid spiked with formaldehyde and acetaldehyde

Repeatability-Across Devices



	Weight Loss	Acetaldehyde	Acrolein	Formaldehyde
	mg/puff block	ug	ug	ug
RSD	33.1%	200.4%	263.2%	129.1%

- Data collected from 20 devices under identical conditions
- Devices 2,6,14,19: Yielded approximately same levels of formaldehyde production
- Overall: Based on the RSD of the 20 devices, TR devices do not seem to be repeatable

Reproducibility-Within Device

Device	Acetaldehyde		Acrolein		Formaldehyde	
	µg/g	% RSD	µg/g	% RSD	µg/g	% RSD
1	64.92	21.8%	8.97	4.8%	269.76	8.2%
2	40.29	29.9%	6.54	8.2%	141.05	16.7%
3	58.16	26.1%	7.58	10.7%	158.60	33.3%
4	90.20	62.3%	9.64	94.3%	258.35	73.0%
5	39.23	18.9%	4.15	17.2%	115.87	7.8%
6	4.43	6.0%	3.59	10.4%	25.41	30.2%



6 devices with 6 replicates per device were collected. Half of these devices were found to be reproducible within a reasonable %RSD (<20%)

Devices gave high variability for native production of aldehydes. Complete characterization of the device and coil is necessary for use as a limited reference product

Transfer of Fortified E-liquids

- Reference liquid spiked with formaldehyde and acetaldehyde
 - Samples were collected at 231 C
 - Data collected from five device tanks and batteries
 - Data presented on a per gram basis
 - This approach has been used in our laboratory to validate trapping capacity and efficiency for new methods

Spiked Reproducibility

Data from the spiked E-liquid collected on the TR devices.

Sample	Yield mg/puff block
1	136
2	281
3	323
4	116
5	266
Average	224
RSD	41.3%

Recoveries of Aldehyde were $\pm 20\%$ of their target value

Results Summary

- Inconsistent production of aldehydes, based on thermal formation, found across the 20 temperature regulated devices tested
- Half of the devices gave acceptable, with-in device, reproducibility of <20%
- Uniform delivery of aldehydes at controlled levels was obtained using a fortified liquid sample

Conclusion

- Temperature regulated devices are not suitable as a reference product for producing analytical levels of native aldehydes
- Full characterization of a single device and coil may be obtained for use as a reasonable laboratory reference product
- Temperature regulated devices were found to be suitable for use as a reference product when using lower temperatures to transfer an e-liquid of known aldehyde concentration

Thank you for your attention